High School Experiment Lab Manual

Introduction

Ammonia

As chemists, we are interested in making ammonia, a chemical composed of one nitrogen atom and three hydrogen atoms. Ammonia is an incredibly important chemical that finds uses in cleaning supplies, smelling salts, and fertilizer. Ammonia-based fertilizers are vital to helping grow the crops which support the global population! Currently, most ammonia comes from something called the Haber-Bosch process. Here are some fun facts about this chemical process:

- Haber-Bosch converts air into the fertilizer needed to feed half of humanity.
- About ½ of the nitrogen atoms in your body originate from ammonia made by Haber-Bosch.
- (Not so fun) fact: Carbon dioxide is also produced in the process of industrial ammonia production.

This last fact shows that Haber Bosch is a major contributor to climate change, which is going to affect our planet in profound ways. It is estimated that billions of people may be displaced from their homes due to climate change over the next 50 years. We are already seeing climate change disasters such as wildfires, hurricanes, and refugees forced to migrate due to dwindling food supply.

Obviously, these impacts of climate change need to be minimized as much as we possibly can. As chemists interested in sustainably producing ammonia, we spend a lot of time thinking about this research question.

What alternative methods can we use to make ammonia?

Scientific Method

Actually, this research question represents the beginning of our entrance into the scientific method. You may have heard of this concept before!

The scientific method starts with a question. This question can come from a variety of places, including observations that we make ourselves or from ideas that we have based on the observations that other chemists around the globe are making.

Some big questions that a scientist might aim to answer using the scientific method include the following:

- How can I make fuel that doesn't pollute the atmosphere when I use it to run my car?
- How can I build a more compact and efficient computer?
- ***What alternative methods can we use to make ammonia?***

The next step of the scientific method is centered upon trying to explain these observations based on our own chemical intuition. This explanation is called a hypothesis.

After we formulate our hypothesis, we finally get to the part where we get to do an experiment. Experiments in chemistry can vary a lot, but they generally involve mixing chemicals together, heating them, or shining a light on them. Once we set up an experiment, it's important to write down all of your observations as changes occur. A few changes that you might see are:

Visual. This can include things like a color change or bubble formation.

Odor. New smells can be a sign that a chemical reaction is happening.

Next, we analyze our results. To do this, we can compare the observations we made to our hypothesis in order to reach a conclusion. If our hypothesis matches our observation, we may have answered an important question, synthesized an exciting new molecule, or discovered a new solution to a big problem. If it doesn't, we can revise our hypothesis/reasoning and perform a modified experiment based on our previous results!

In this sense, the scientific method rarely ever "ends". There's always more to learn from our experiments, which gives us a brilliant opportunity to continue the cycle by proposing more hypotheses based on the results from our first experiment!

Forming Our Hypothesis

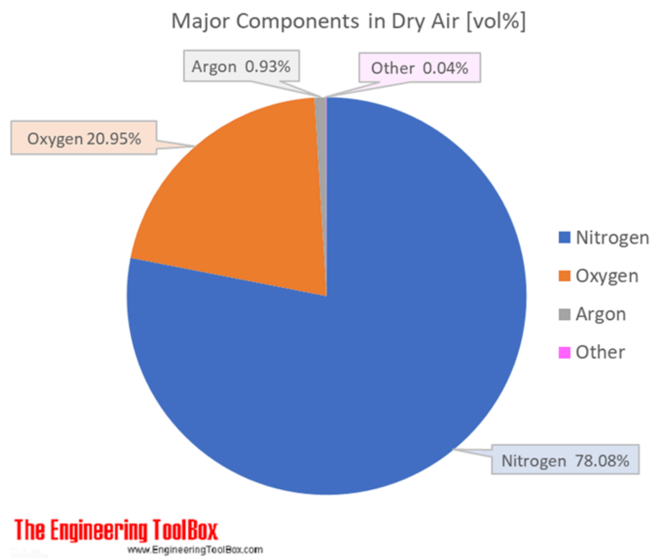
Now that we have solidified our research question, let's formulate a hypothesis.

As a reminder, our question is:

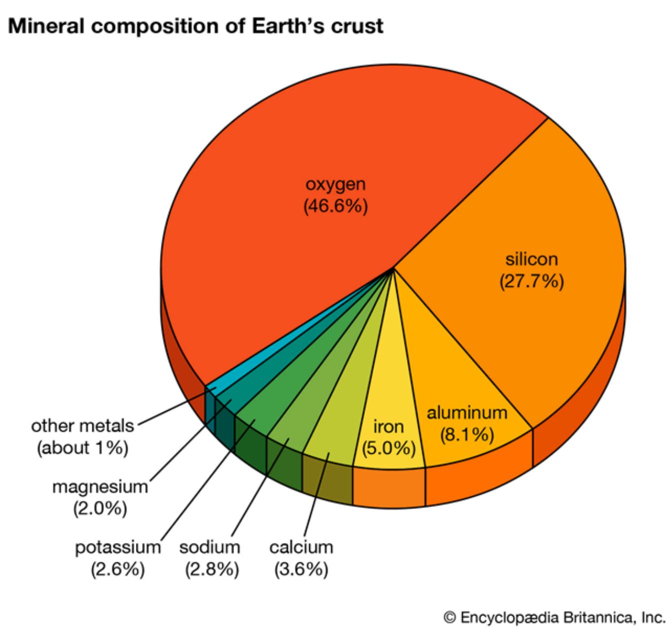
What alternative methods can we use to make ammonia?

As mentioned before, one major goal we have in studying this question is choosing ways to make ammonia which are sustainable. That means choosing compounds which are plentiful and do not produce environmentally harmful products when we have them undergo reactivity to make ammonia. Let's think about some abundant chemicals on Earth to decide where we can get our nitrogen and hydrogen:

The first is air. Here's a pie chart which shows the composition of Earth's atmosphere. As you can see most is nitrogen, though oxygen and argon also represent a significant percentage as well.



The next is dirt, particularly in the Earth's crust. Here's another pie chart for this natural resource. Most of it is oxygen, with other components being elements such as silicon, aluminum, and iron.



Finally, there are other resources that are available. These include plants and water. Water is frequently denoted with the chemical formula H_2O. This means that it contains two hydrogen atoms (H) and one oxygen atom (O).

Does anyone see a source of nitrogen and hydrogen that we can use for making ammonia?

As you can see, nitrogen is a major component of air and hydrogen is a major component of water! These two both could be useful in making ammonia. In fact, one major source of ammonia in nature comes from lightning strikes providing electrical energy necessary for ammonia production from air and water.

It is useful to note that organisms are capable of making ammonia from natural resources as well! Plants sometimes have bacteria growing in their roots which make ammonia that helps support the plants during their life.

Now let’s revisit our question again. We’ve agreed that air and water are fantastic candidates for making ammonia. Now the question is how we can get them to react.

What source of energy might we be able to use?

As mentioned before, lightning is a source of electrical energy which produces ammonia in nature. It’s impractical to summon lightning on command to do this reaction, so let’s think about how we can supply electrical energy to a system to make ammonia.

A battery is a fantastic option for this! Now that we have all of the components we need, let’s write down a possible answer to our research question. This will be our hypothesis.

Electrochemistry

Today, the source of electrical energy we will use to try to generate ammonia will be a 9V battery. Before we move to the experiment, let us first consider how a battery works:

An electrochemical cell moves electrons from one electrode (the anode) to another electrode (the cathode).

This flow of electrons can be used to perform work, such as powering a lightbulb or your TV remote.

The flow of electrons can also cause special chemical reactions to happen.

However, to move these electrons in our reaction, we need energy! By harnessing this energy, we can imagine doing the following chemical reactions which would generate ammonia:

At the anode, we can do an electrochemical reaction with water to yield electrons and protons.

Since this reaction involves the production of electrons, it is known as an oxidation reaction.

At the cathode, N_2 is waiting to receive those protons and electrons.

Since this reaction requires electrons to go, it is known as a reduction reaction.

Constructing the Electrolysis Setup

Now that the necessary background is in place and you have a hypothesis to test, you can finally run the experiment. The materials you will need are as follows:

- Colorless plastic cups (2 – one large and one small)
- Syringes (2)
- Flat Metal thumbtacks
- Epsom Salt
- 9V battery
- Cardstock
- Paper towels
- Scissors (not in kit)
- Water (not in kit)
- Marker/Sharpie/writing utensil (not in kit)

Safety note: Before doing any science experiment, it is vital that possible hazards are considered. This ensures that what we are doing is safe!

- Since batteries provide electrical energy, they can pose a risk of electric shock. To avoid this, try not to touch both terminals of the battery at once! However, the amount of electric current produced by a 9V battery is not enough to be extremely dangerous.
- Another important note is that batteries can sometimes leak, and it's important to avoid contact with any leaking batteries. If your battery is leaking, find a different one!
- Minimize contact of any chemicals that you handle. This means avoiding consumption of our solution, as well as the salt being dissolved in water. Again, the chemicals that you'll use in this experiment are not incredibly dangerous, BUT any compound in an experiment should be handled with caution.

1. Stack the small cup on top of the 9V battery. Center the battery underneath the cup and use the marker to note where the battery terminals are.
2. Remove the stacked plastic cup and puncture the markings with two thumbtacks. The sharp ends of the thumbtacks should sit inside the cup. Use a marker to push the cup down onto the thumbtacks to avoid poking yourself. Make sure that the thumbtacks do not touch, but are close enough together to touch both terminals of the 9V battery. Label the cup "Electrolysis Cell – $MgSO_4$ in H_2O".
3. Take your piece of cardstock and center the battery on top of it. Mark the two terminals on the piece of cardstock. Use your pencil to puncture through the cardstock to create two holes at the terminal sites; these will end up being about the same size as our syringes.

4. Carefully trim off the narrower ends of both syringes using a pair of scissors (the plastic may be difficult to cut, so go slowly!). Push the syringes through the holes in the card stock, to about the 1.0 mL line and set aside.
5. Add approximately 15 mL (1 Tbsp) of Epsom salt ($MgSO_4$) to the large cup followed by 120 mL (1 cup) of water. Stir the solution until the salt dissolves in the water.
6. Pour the electrolyte solution into the small cup containing your thumbtacks until the cup is about ¾ full. Discard the rest of the electrolyte solution (the drain is fine because the solution is just $MgSO_4$ which is non-toxic and water) and dry out the cup.
7. Next, place the cardstock/syringe apparatus over the cup and line up the syringes over the top of the thumbtacks. Pull solution up into the syringes to the 0.8 – 0.9 mL mark, making sure to avoid getting any bubbles in your syringe. If you do get bubbles, push the plunger all the way down and draw up fresh solution. You may need to repeat this a few times to get rid of all the bubbles. Push the syringes down until they cover the sharp ends of the thumbtacks.
8. Now take the 9 V battery and place in the bottom of the large (and dry!) cup and pack around it with paper towels to snugly place the battery in the middle of the cup. Notify the teacher if the battery is leaking/corroded as battery acid is corrosive.

Running the Electrolysis

1. Record the liquid level in the syringes at both the (–) and (+) terminals on your worksheet.
2. Having made our hypotheses, stack the two cups, connecting the thumbtacks to the two terminals on the 9V battery. Write down your observations of what happens on your worksheet.
3. After approximately 1-2 minutes, or when you can clearly measure a large difference in the water level in each syringe. Record any changes on your worksheet.

Now that you've run the experiment, think about whether any of the gases in the bubbles we saw forming were ammonia. First, let's balance the reactions that need to be happening in for ammonia production to be occurring:

First up is water oxidation to protons and oxygen. Who can balance this equation by filling in the blanks?

- Water Oxidation: $\_\_\ H_2O \rightarrow 3/2\ O_2 + \_\_\ H^+ + 6\ e^-$

Next is nitrogen reduction to ammonia with protons and nitrogen from air. Who can balance this equation by filling in the blanks?

- Nitrogen Reduction: $N_2 + \_\_\ H^+ + 6\ e^- \rightarrow 2\ NH_3$

Now, let's add these reactions together to get the overall chemical reaction that we're seeing in our electrochemical cell! What reaction do you get as a result of this addition?

- Balanced reaction: $\_\_\ H_2O + \_\_\ N_2 \rightarrow \_\_\ O_2 + \_\_\ NH_3$

Now that we have this overall reaction, we can finally assess the validity of our hypothesis by determining if we made ammonia. If this oxidation and reduction reaction were indeed happening, what would the ratio of gases evolved at the two electrodes be?

Hint: look at the coefficients on the gaseous products in the overall reaction.

- Expected ratio of gas:_____

If this reaction were indeed occurring, you might expect a gas ratio of 4:3 in the two syringes. This is probably not the ratio that you saw! What is the approximate ratio of gases that were formed at the two electrodes? Does this ratio suggest that ammonia is being evolved?

It doesn't! Does this result confirm your hypothesis?

Because this result is potentially unexpected, it's important that we try to explain what actually happened. What other molecules are present that may have reacted instead of nitrogen?

Protons!

Proton reduction could have been occurring instead of nitrogen reduction! This is what that reaction looks like.

- Proton Reduction: $6\ H^+ + 6\ e^- \rightarrow 3\ H_2$

Would a gas have been formed from this reaction? Which one?

Hydrogen!

Hydrogen would have been formed. Now take a look at the overall reaction assuming that proton reduction were occurring instead of nitrogen reduction.

What ratio of gases would you have expected?

- Balanced reaction: $2\ H_2O \rightarrow O_2 + 2\ H_2$

2:1!

Now let's look at these two potential overall reactions side by side. Do you stand by your original hypothesis?

Another added piece of knowledge which could help in your decision is that ammonia is very water soluble. Hydrogen and oxygen are not quite as water soluble. Does this affect your reflection on your hypothesis?

Conclusions

Now that you've studied your hypothesis, you can move to the last segment of the scientific method where we figure out what to do with our new knowledge! We also want to revisit the experiment we did to see if we can potentially envision other ways of answering our guiding question.

We didn't make ammonia using a 9V battery today, but we did make hydrogen!

This is an interesting result, because hydrogen is a clean, carbon-free fuel. While this may not solve our specific question, this result may nevertheless be interesting for its potential application in fuel cells!

It's important to know that in science, this experimental outcome is quite common and acceptable. As long as you made sure that your experiment tested your original hypothesis, you learned something anyway. This is the most important part of following the scientific method—learning something about your original research question.

Finally, let's think about what we might do differently based on what we learned today. If we were to do this experiment again, what could we change to try to lead to a different result?

- Trying a different solvent than water!
- Trying a different material than a thumbtack as our electrode!
- Trying to do the same experiment in an atmosphere of 100% nitrogen!